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Sugarcane Wax Studies, 1942-1943*

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Investigational work dealing with sugarcane wax was continued through the year. During the cane harvesting season particular attention was given to (a) the wax content of different varieties of sugarcane, (b) the effect of burning cane on its wax content, (c) a further study of the removal of wax from the cane in milling operations, and (d) the wax content of mud press cake from various factories. Pilot plant studies, dealing with the extraction and purification of sugarcane wax and other products, were conducted during the remainder of the year. Each phase of the work will be discussed briefly in this paper. The reader should consult the two previous reports (1, 2) in order to obtain a better understanding of the project as a whole.

Wax Content of Different Varieties of Sugarcane

Early in this work Fort (3, 4) pointed out the existence of a varietal factor in the wax content of Louisiana sugarcane. This subject was given further study and a new technic for determining crude wax in sugarcane was developed which, it is believed, is an improvement over the direct solvent extraction procedure previously employed. Briefly, the method is as follows:

A sample of finely divided sugarcane is pressed in a hydraulic press under conditions which extract between 80 and 85% of the juice. The bagasse briquet is weighed and analyzed for moisture and wax content. The wax is calculated to original cane basis. The juice is analyzed for its wax content by a combined trapping and solvent-extraction method similar to the method described by Bardorf (5). Calcium hydroxide, calcium phosphate and lead acetate with or without added filter-aid were tried for trapping the wax. Each was equally effective but as lead acetate gave visual demonstration of effective clarification, this reagent was used in this study. The ordinary basic lead acetate used for sugar analyses was found satisfactory. The lead precipitate carrying the trapped crude wax is filtered into a Gooch crucible and is washed with water at room temperature until approximately freed of water-soluble substances. It is then dried and the wax extracted in the usual manner. A record is made of the amount of juice extracted from a given weight of cane in order to calculate the wax found in an aliquot portion of the juice to cane basis. With the type of hydraulic press employed (cage) most of the wax remained with the cake of bagasse rather than becoming dispersed in the juice as happens in commercial milling. For results of reasonable accuracy, it would be possible to dispense with the determination of wax in the juice, in which case a constant correction for the amount of wax in the juice would be added to the wax found in the bagasse. Each analyst should determine this correction as it would vary with the type of equipment and technic involved.

The wax content of different varieties of sugarcane determined by the method described above is given in Table 1. The data relating to the proportion of hard wax and fatty matter are based upon the fractionation of the crude wax extracted from the bagasse samples only. The amount of wax involved in the trapped material from the juices was so small as to make its fractionation unreliable. Even if the proportion of fats to hard wax were not the same from the two sources, the data reported would not be greatly in error. There seems to be almost as much variation between samples of a single variety as between samples of different varieties, the reasons for which are not well understood. Doubtless there are cultural factors not yet evaluated that greatly influence the amount of wax deposited on the cane stalks. There is a definite tendency, however, for certain varieties to have a high wax content and others to have a low wax content. Of the commercial varieties grown in Louisiana only C. P. 29/116 cane definitely has a low wax content. This verifies results obtained by Fort (4). Co. 290 cane appears to have about the highest hard wax content and the lowest fat content, with the possible exception of C. P. 807 which is scarcely grown any longer. The predominence of Co. 290 cane over other varieties grown in the western part of the Louisiana sugar district may be responsible, in a large measure, for the fact that the press cake from the factories located in the western section invariably contains a higher crude wax and a higher proportion of hard wax than press cake from factories located in the eastern section. Based upon the analysis of sugarcane, there is a potential yield of about 18 million pounds of crude wax from an annual 5 million-ton Louisiana sugarcane crop. Unfortunately, however, only about 40% of this wax, or a little over 7 million pounds, is obtainable from the waste clarification muds; the remainder of the wax is discarded with the bagasse, and it cannot be recovered profitably. This figure of 7 million pounds agrees well with that calculated from the mud press cake if one assumes a production of 16 pounds of dry muds per tone of cane processed and an average crude wax content of 9% in the dry mud.

Wax Content of Certain Products of Milling

In order to supplement information given in last year's report (2) regarding the distribution of wax in various factory products, particularly in juices and bagasse, a study was made of the wax content of these products from a single known variety (Co. 281) resulting from experimental milling experiments carried out under carefully controlled conditions.

In these tests, which were run in duplicate, 80 pounds of Co. 281 cane, thoroughly cleaned of trash, were put through an experimental 3-roller mill 5 times. Dry milling was used for the first two extractions and 16 pounds of water was added as imbibition water in the last three millings. The juice from the first milling (crusher juice) was weighed and analyzed separately from the remaining juices which were composited prior to sampling and analyzing. The bagasse was also weighed and analyzed. The results obtained in this series of tests are given in Table 2. It should be noted that the proportion of wax which became dispersed in the juice (41% of the total) is about the same as was found under commercial milling practices in spite of the fact that cold imbibition water was used in these tests. It may be concluded that the temperature of the imbibition water as usually employed has no pronounced effect upon the removal of the wax from cane. It is very likely that the mass of cane would have to be heated above the melting point of the wax before the benefit of a high water temperature would be noticeable.

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Table 1. Wax Content of Millable Cane (1942 Crop from Houma Station)

Variety of Cane	Number of Samples	Crude Wax Min.	Crude Wax Max.	Crude Wax Ave.	Hard Wax on Cane	Hard Wax on C. Wax	Fatty Fraction on Cane	Fatty Fraction on C. Wax
		%	%	%	%	%	%	%
C. P. 807	1	--	--	0.241	0.165	68.6	0.076	31.4
Co. 290	9	0.167	0.233	0.206	0.137	66.3	0.069	33.7
Co. 281	7	0.131	0.231	0.176	0.098	55.5	0.088	44.5
C. P. 28/11	1	--	--	0.186	0.109	58.5	0.077	41.5
C. P. 28/19	5	0.154	0.245	0.213	0.118	55.6	0.095	44.4
C. P. 29/103	5	0.162	0.190	0.176	0.099	56.2	0.077	43.8
C. P. 29/116	6	0.100	0.127	0.114	0.058	51.0	0.046	49.0
C. P. 29/120	4	0.174	0.193	0.186	0.101	54.4	0.085	45.6
C. P. 29/320	4	0.163	0.178	0.172	0.098	56.7	0.074	43.3
C. P. 34/120	4	0.122	0.185	0.163	0.091	55.8	0.072	44.2

Table 2. Wax Removal fron Sugarcane by Milling—Co. 281 Cane

Test Number	Crude Wax Content of Mill Products: Crusher Juice on juice	Crusher Juice on cane	Remaining Juices on juice	Remaining Juices on cane	Bagasse on sample*	Bagasse on cane	Total Crude Wax	Wax Removed by Milling
	%	%	%	%	%	%	%	%
1	0.044	0.022	0.064	0.023	0.576	0.086	0.131	34
1a	0.054	0.025	0.073	0.030	0.550	0.076	0.131	42
2	0.061	0.040	0.073	0.020	0.602	0.085	0.145	41
2a	0.070	0.045	0.088	0.025	0.564	0.080	0.150	47
3	0.076	0.048	0.062	0.019	0.798	0.118	0.185	41
3a	0.059	0.036	0.081	0.024	0.520	0.073	0.133	45
Average		0.036		0.024		0.086	0.146	41

* Dry weight.

Table 3.
Effect of Trash Removal by Burning on Crude Wax Content of Sugarcane

Variety of Cane	Number of Samples	Crude Wax Content: Normal Cane	Burned Cane	Change: Actual	Change: Percent
		%	%	%	
Co. 290	3	0.226	0.206	—0.020	—9.0
Co. 281	1	0.191	0.176	—0.015	—7.8
C.P. 28/19	1	0.245	0.234	—0.011	—4.5
C.P. 29/116	1	0.117	0.244	+0.127	+108
C.P. 36/70	1	0.164	0.207	+0.043	+26

The Effect of Burning Trash on the Wax Content of Cane

It has been claimed by Clacher (6) that burning cane for the purpose of removing trash—cane leaves, etc.—lowers the wax content of sugar factory press cake from about 14 to 10%. The experiments reported in Table 3 are not sufficiently complete to confirm or refute this claim since the cane was not carried through factory processing. An attempt was made, however, to determine how the crude wax of sugarcane was affected by the method of burning adopted by the Louisiana sugar industry, namely, by firing small piles of trashy cane laid across the ridges after the green leaves have become withered.

Table 4.
Wax Content of Clarification Mud Press Cake—1942 Season.

District	Factory	Number of Samples	Crude Wax-Moist.-free cake Ave. Hard Wax.: Min.	Max.	Ave.	On Sample	On C. Wax	Ave. Acetone Soluble: On Sample	On C. Wax
			%	%	%	%	%	%	%
Eastern	1	5	6.78	9.70	8.70	5.90	67.87	2.80	32.13
	2	6	8.19	12.39	9.69	6.19	63.88	3.50	36.12
	3	2	9.34	12.90	11.12	8.13	73.09	2.99	26.91
	4	3	5.94	9.63	7.99	5.32	66.63	2.67	33.37
	5	5	5.83	8.39	7.47	5.17	69.16	2.30	30.84
	6	8	5.85	11.09	9.26	6.04	65.21	3.22	34.79
	7	3	3.26	7.38	5.32	3.67	69.03	1.65	30.97
	8	6	6.78	9.77	8.70	5.87	67.48	2.83	32.52
	9	5	5.30	9.42	7.21	4.48	62.20	2.73	37.80
	10	9	7.92	13.69	10.33	6.94	67.19	3.39	32.81
	11	4	5.99	11.08	7.86	5.55	70.63	2.31	29.37
	12	1	--	--	10.58	7.50	70.92	3.08	29.08
	Weighted Average				8.75	5.86	66.97	2.89	33.03
Western	1	4	10.54	12.59	11.23	8.42	74.98	2.81	25.02
	2	9	14.08	18.90	16.02	11.92	74.39	4.10	25.61
	3	6	8.98	15.58	13.19	10.11	76.68	3.08	23.32
	4	3	10.06	13.82	13.04	10.01	76.75	3.03	23.25
	5	3	14.02	14.87	14.47	10.53	72.78	3.94	27.22
	6	4	9.27	12.88	10.88	8.35	76.71	2.53	23.29
	7	Composite	--	--	12.83	9.67	75.38	3.16	24.62
	Weighted Average				13.57	10.22	75.31	3.35	24.69

Table 5.
Summary of Wax Content of Louisiana Clarification Mud Press Cake.
(Moisture-free basis)

District	Season	Number of Samples	Crude Wax	Hard Wax	Acetone Sol. Fract. On Mud	On C. Wax.
			%	%	%	%
Eastern	1940	7	6.99	4.47	2.52	35.79
	1941	39	9.19	6.32	2.87	31.23
	1942	57	8.75	5.86	2.89	33.03
		Average	8.80	5.94	2.86	33.35
Western	1940	9	12.77	9.76	3.01	23.57
	1941	84	11.89	9.09	2.80	23.55
	1942	30	13.57	10.22	3.35	24.66
		Average	12.36	9.41	2.95	23.93

Table 6.
Deterioration of Mud Press Cake.
(Moisture-free basis)

Source	Storage Period	Storage Condition	Crude Wax Content	Crude Wax Loss	Hard Wax Content	Hard Wax Loss	Acetone-sol. Fract. Content	Acetone-sol. Fract. Loss
	weeks		%	%	%	%	%	%
Factory 1	0	--	16.51	--	12.72	--	3.79	--
	4	wet	13.27	19.6	11.65	8.4	1.62	57.2
	8	wet	11.91	27.9	10.07	20.8	1.84	51.5
	12	wet	10.01	39.4	8.41	33.9	1.60	57.8
Factory 2	0	----	7.50	--	5.12	--	2.38	--
	4	wet	7.38	16.0	4.97	2.9	2.41	-1.3
	8	wet	5.24	30.1	3.69	27.9	1.55	34.9
	12	wet	4.16	44.5	3.00	41.4	1.16	51.3
Factory 3	0	----	12.71	--	8.90	--	3.81	--
	4	wet	11.99	5.7	9.22	-3.6	2.77	27.3
	8	wet	10.39	18.7	8.76	1.6	1.63	57.2
	12	wet	10.56	16.9	8.47	4.8	2.09	45.1
Factory 4	0	----	17.36	--	13.87	--	3.49	--
	4	wet	14.24	18.0	11.87	14.4	2.37	32.1
	8	wet	14.70	15.3	12.55	9.5	2.15	38.4
	12	wet	15.17	12.6	12.46	10.2	2.71	22.3
Average	0	----	13.12	--	9.46	--	3.66	--
6 samples	30	air dry	12.36	5.8	8.38	11.4	3.98	-8.7

Six stalk samples were taken from individual heaps before and after firing and they were further cleaned by hand prior to grinding, pressing and analysis. Judging from the results obtained, which are summarized in Table 3, the experimental errors were rather large, for in some instances firing apparently decreased the amount of wax found while the reverse was the case with other samples. It appears that burning the trash did not destroy much, if any, of the wax, but inspection clearly indicated considerable melting of the wax. It is conceivable that after the wax had been melted it will adhere to the rind more tenaciously than unmelted wax during the milling operations. If this occurs the wax content of the mud press cake is certain to be lowered. Further study of this question should be made, for it has very practical significance. It was noted that burned cane always yielded darker crude waxes than unburned cane which means that burning adds to the color problem.

Wax Content of Sugar Factory Mud Press Cake

Through the co-operation of sugar-factory operators samples of clarification mud press cake were submitted for analysis at about weekly intervals throughout the season. The method for determining wax was the same as used in previous seasons with the exception that benzene was used instead of carbon tetrachloride for making the extractions. The results, summarized in Tables 4 and 5, are expressed on the moisture-free basis.

The results show that the wax content of the mud press cake from the factories located west of the Atchafalaya River is appreciably higher than the wax content of the press cake from the factories locatea east of this river. It may be, as already stated, that this difference is due principally to the fact that the western factories process a considerably larger proportion of Co. 290 cane than do the eastern factories. This fact would also explain the much higher proportion of hard wax found in the presscake from the factories located west of the river. (See Table 1.)

From these results, it is obvious that the best source of wax from the standpoint of commercial extraction would be the press cake from the group of factories located in the western section of the sugar-growing area of Louisiana. Since there are a few factories located in the eastern section of the area that produce press cake having a relatively high wax content it leads one to wonder why this occurs. It is quite likely that more light would be thrown on this question if the actual weight (dry) of the mud press cake from different factories were available. Unfortunately, in most cases the wet weight of press cake is only estimated and the moisture never determined. It is known, however, that many factors influence the weight of clarification muds as the weather, variety of cane, soil, fertilizer, milling and clarification practices, and each in turn would affect the wax content of the clarification mud press cake. There is need for considerable study of factors that affect the wax content of the press cake for it may be possible to increase the wax content materially without introducing deleterious effects in factory operation, thereby increasing the value of the press cake as a source of valuable by-products.

Deterioration of Mud Press Cake

It has long been known (7, 8) that when clarification muds are allowed to ferment the wax content decreases upon aging and that the hardness of the crude wax improves. This indicates that the fats are consumed by the microorganisms more rapidly than the wax. Since no data were available, some preliminary results were obtained by analyzing 4 samples of muds periodically after being stored under wet conditions as might be met if the muds were placed in a pile and allowed to weather. The summarized results are given in Table 6.

The data do not seem to be as consistent as one might expect but this is doubtless due to faulty sampling of the mass which had undergone uneven deterioration. It is evident, nevertheless, that wet muds deteriorate with considerable rapidity in respect to both the hard wax and the fatty constituents. For some reason, it would appear that mud cake from plate presses deteriorates at a more rapid rate than mud press cake from vacuum filters. (Compare factory samples 1 and 2 with 3 and 4.) It is possible that more complete removal of sugars and water-soluble matter, which usually occurs in the operation of the vacuum filters, causes the activity of microorganisms to be lessened or changed in some manner which results in a lower rate of wax and fat destruction.

As might be expected, drying freshly produced muds greatly reduces the rate of deterioration. One might have expected greater stability than has been found to be the case if the average analysis of 6 samples that had been stored for 30 weeks is indicative. These samples showed a loss of almost 6% in the crude wax content and a possible transformation of some of the waxy constituents into acetone soluble matter with a loss of hard wax and a gain of fatty material.

Pilot Plant Studies

A number of phases of wax extraction were studied during the year. Those of most importance were concerned with (a) crude wax extraction as affected by the type of solvent, the temperature of extraction and the physical condition of the mud press cake particles; (b) the fractionation of the crude wax into its fatty and wax components by different solvents; (c) the relationship between the type of solvents used for crude wax extraction and refining operations and the physical and chemical properties of the hard wax produced from different sources; and (d) methods of refining the dark hard wax to produce wax of light color which might enhance its value for special applications.

Table 7.—Comparison of Solvents as Crude Wax Extractant.
(75° to 80°C, 2½-hour extraction cycle)

Source of Muds	Toluene				Naptha-Shell 8220, 2220				Benzene			
	No. of runs	Weight charge-Muds	Solvent/muds	Extraction efficiency	No. of runs	Weight charge-Muds	Solvent/muds	Extraction efficiency	No. of runs	Weight charge-Muds	Solvent/muds	Extraction efficiency
		Lbs.		%		Lbs.		%		Lbs.		%
1-1941	31	15.0	10.8	91.1	11	17.7	8.3	91.4	27	17.5	10.0	91.4
2-1941	31	17.3	9.7	85.3	20	19.1	7.6	86.4	6	19.7	8.6	83.9
					Naptha-Skellysolve "C"							
1-1941					23	17.8	7.9	89.7				
	Toluene				Naptha-Shell 8196							
1-1942	16	17.6	9.3	85.8	12	16.7	8.4	84.1				
2-1942	14	18.3	9.1	83.4	4	18.8	7.1	88.0				
3-1942	5	27.2	5.5	82.1	--	--	--	--				
4-1942	8	25.4	6.0	86.0	10	27.8	4.8	88.9				
5-1942	8	44.2	3.5	83.6	3	44.0	3.2	86.7				
6-1941	19	27.9	5.3	82.9	11	27.1	5.0	83.2				
7-1942	--	--	--	--	3	27.7	5.3	87.1				

In last year's report (2) a comparison was made of different solvents for the extraction of crude wax. These included toluene, benzene and a petroleum naphtha. Since then, further tests have been made with toluene and with two additional types of petroleum naphtha. The technic involved was the same with the exception that the solvent used for determining wax in the original muds and in the extracted muds was the same as was being used for making the pilot plant extractions. As various solvents yield different wax values, it was felt that more accurate measurement of extraction efficiencies would be obtained if the same solvent was used for both purposes rather than different solvents.

The results obtained with the two additional types of naphthas are not sufficiently different to warrant extended discussion. A summary of results is given in Table 7 of all the extraction work for the past

method of handling the product was to hydrolyze the fats in an acetone solution with barium hydroxide, as the first step. Such an operation may well be combined with hard wax production since, if acetone is employed to remove the fatty matter from crude wax, all that needs to be done would be to concentrate the acetone solution of fats to the desired concentration and proceed with the barium hydroxide saponification process. The barium soaps are insoluble in acetone (but not in 2-butanone) and may thus be easily separated from the acetone solution of the liberated alcohols, sterols, hydrocarbons, etc. Recovery of the fatty acids is possible by decomposing the barium soap with a mineral acid. This may be done in either an acetone solution, in which case the barium salt may be recovered in crystalline form (such as barium chloride), or as a water suspension in which case the fatty acids rise to the surface and solidify upon cooling, making their removal possible. Purification of some of the fatty in this fraction unless destroyed by the process used to fractionate the fatty matter.

Uses for Sugarcane Wax

It has long been known that sugarcane wax can be used for a large number of applications replacing, in part or wholly, other commercial waxes. A large number of concerns have tested for various applications the wax resulting from these and previous experiments. As might be expected, opinions as to the value of the wax varied considerably, even in the same fields of applications. Inasmuch as there is a considerable loss of wax involved in obtaining a light-colored wax, the main application of the wax is expected to be in those fields where the dark color of the hard wax would not interfere. It has been demonstrated conclusively by the trade that sugarcane wax can be used successfully and it is believed that a definite market for the wax is awaiting commercial production.

Table 10.—Some Properties of Hard Sugarcane Waxes of Louisiana Origin

Source of Wax	Solvents for		Hard Wax soluble in-			Colorimeter Readings			Ash	Specific Gravity
	Cr. Wax	Fats	Acetone	EtOH*	Total	510	560	610		
			%	%	%				%	
1-1942	Toluene	Acetone	68.2	13.3	81.5	27.6	22.5	22.3	2.39	1.000
	Toluene	Butanone	65.0	13.1	78.1	23.2	17.7	15.6	1.95	
	Naptha	Acetone	58.8	8.5	67.3	22.5	16.9	15.2	1.98	
	Naptha	Butanone	62.0	8.2	70.2	17.4	13.1	12.0	1.60	
2-1942	Toluene	Acetone	63.7	10.5	74.2	18.4	13.8	12.1	2.51	1.011
	Toluene	Butanone	63.2	13.8	77.0	25.2	18.7	16.5	2.40	
	Naptha	Acetone	62.0	11.4	73.4	12.1	9.5	8.7	2.44	
3-1942	Toluene	Acetone	66.7	7.5	74.2	26.1	21.7	23.1	3.97	
4-1942	Toluene	Acetone	68.5	13.5	82.0	20.3	16.8	17.3	2.69	
	Toluene	Butanone	68.8	12.7	81.5	17.0	13.7	13.3	2.18	
	Napthe	Acetone	64.2	11.3	75.5	16.8	12.4	10.7	2.40	
	Naptha	Butanone	60.3	9.9	70.2	12.5	8.6	8.0	1.89	
5-1942	Toluene	Acetone	62.2	12.5	74.7	25.5	20.9	20.6	2.97	0.999
	Toluene	Butanone	69.9	10.0	79.9	21.4	16.7	15.1	2.84	
	Naptha	Acetone	67.1	12.0	79.1	14.3	11.5	11.4	2.85	
6-1941	Toluene	Acetone	66.2	11.4	77.6	21.0	16.7	17.0	1.53	0.996
	Toluene	Butanone	67.7	11.4	79.1	20.7	16.6	16.3	1.58	
	Napthe	Acetone	64.0	8.3	72.3	16.8	12.6	13.2	1.72	
	Naptha	Butanone	60.7	8.5	69.2	16.3	11.4	11.2	1.16	
7-1942	Naptha	Butanone	65.9	6.9	72.8	13.0	10.2	9.8	1.05	
Averages	Toluene	Acetone	65.9	11.4	77.3	23.1	18.7	18.7	2.68	1.0015
	Toluene	Butanone	66.9	12.2	79.1	21.5	16.7	15.4	2.19	
	Naptha	Acetone	63.2	10.3	73.5	16.5	12.6	11.8	2.28	
	Naptha	Butanone	62.2	8.4	70.6	14.8	10.8	10.3	1.43	

* Ethanol extraction followed acetone extraction.

acids may be accomplished by vacuum distillation as is now done commercially. The fate of the chlorophyll that accompanies the barium soap in such a process has not been determined, but it may be possible to recover it from the residues after the fatty acid distillation. Almost quantitative recovery of the sterols is possible by evaporating the acetone solution resulting after the saponification process until the water concentration reaches about 20% (about half the original volume) and then chilling. The crystalline material may be recovered by filtration or other physical means. If the mother liquor is concentrated still further in order to recover the acetone and decrease its concentration in the solution, another product may be obtained. The exact nature of this product has not yet been determined but if any carotene were present in the fatty product, and there is evidence that some is present, it will be concentrated

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two years. It can be stated with certainty that petroleum naphthas boiling within the range of about 85° to 120° C. can be used successfully for the extraction of crude wax. They have a distinct advantage over toluene in their lower cost, specific gravity and solvent power toward some of the pigments encountered. Although there did not appear to be any great difference between toluene and the naphthas in regard to the amount of crude wax extracted under pilot plant conditions, about 10% more extractables were obtained from muds when toluene was used in comparison with the napthas in the analytical determination of crude wax. It is evident from this that somewhat different constituents are extracted by different solvents. This is partly verified in some experiments dealing with the fractionation of the hard wax with certain solvents which will be discussed later. There is evidence that after continued use the naphthas gradually undergo a change which prevents complete volatilization under the conditions used in the pilot plant operations, with the result that in extreme cases, the hard wax solidifies with a crystalline structure rather than with a uniformly amorphous structure. It seems that the petroleum naphtha which contained aromatic hydrocarbons was worse in this respect than the straight-chain hydrocarbon naphtha. Whether longer steaming of the wax would correct this condition was not determined. The wax is relatively easy to free from either benzene or toluene with live steam in comparison with the petroleum naphthas after they have been in use for some time.

The rate of extracting wax from clarification muds by a given procedure is noticeably affected by the temperature, up to a certain point, at which the extraction is made. Since wax is not very soluble at ordinary temperatures in most solvents that would be used in commercial work, but is soluble in all portions at temperatures above the melting point of the wax, it is obvious that the optimum temperature for extraction should lie close to or above the melting point of the wax. The melting point of crude wax varies with the proportion of fatty matter present but normally it is close to 60° C. In a series of tests, reported in Table 8, where the effect of temperature was determined on muds from a single source using toluene and a petroleum naphtha, it was found that temperatures of 60° or lower were distinctly inferior to temperatures of 70 to 80° C., and that the proportion of wax extracted was not much improved by increasing the temperature above 70° C. for naphtha or above 80° C. for toluene. The amount of wax extracted was not appreciably increased by increasing the time cycle from 2 to 2½ hours, indicating that the shorter period had already exceeded the optimum for the type of mud press cake being extracted.

Table 8.

Effect of Temperature upon Crude Wax Extraction of Mud Press Cake.

Source of Muds	Solvent	Extraction Efficiency* at 50°C	60°C	70°	80°	90°	Extn. Cycle
		%	%	%	%	%	hrs.
1-1941	Toluene	81.1	84.9	85.8	88.9	87.6	2½
1-1941	Naptha	86.6	87.7	88.9	90.8	--	2½
		85.6	85.5	90.4	90.3	--	2

* Results expressed on total wax present.

The rate of extracting crude wax from press cake under optimum temperature conditions is governed by the physical condition of the material being extracted. Theoretically most rapid extraction would be obtained from pulverized material but this was not verified because it was not possible to use pulverized material at all satisfactorily in the process that was adopted in the pilot plant. It was demonstrated, however, that porous mud press cake material which contains a large proportion of fibrous matter (intentionally added where the muds are handled with the continuous vacuum filters) offered no appreciable resistance toward extraction. In other words, equilibrium between the solvent and wax was quickly established. Conversely, the lower the fiber content of the mud press cake, the slower was the rate of wax extraction because the particles dried with a rather impervious structure.

The density of the mud press cake also varies with the fiber content. Since cane fiber has a very much lower density than the lime precipitate from the juice clarification process, it is to be expected that the press cake from the vacuum filters would have a much lower density than muds from the old type plate presses. What might be termed normal variations in the density of mud press cake of Louisiana sugar factories after drying may be observed in the data given in Table 7 under the heading of "Weight charge-Muds". In operating the extraction process, the disgester was filled to about the same level each time so that the variation in weight is a measure of the density of the press cake. Calculated from the volume of material put in the digester (not shown in Table) the density ranged from 11 to 32 pounds per cubic foot. This fact will have to be considered in designing a commercial wax-extraction plant.

Removal of the fatty matter from crude wax to yield a hard wax may be accomplished in several ways as stated in previous reports. In this study the results of which are given in Table 9, two solvents were compared quite critically—acetone and 2-butanone (methyl ethyl ketone). The latter solvent is more efficient than acetone in removing the fatty matter and pigments but, it also has a higher solvent power on wax constituents. About 5% more hard wax remains after the treatment with acetone, regardless of whether the crude wax was originally extracted from the mud press cake with toluene or with a petroleum naphtha. It is also interesting to note that a greater yield of hard wax is derived from naphtha-extracted crude wax than from toluene-extracted crude wax when either acetone or 2-butanone is employed for removing the fatty fraction.

These results might lead one to think that the composition of the wax would vary with the solvent used for extracting the crude wax, as well as with the solvent used for separating the fatty matter from the hard wax. This is not borne out in full, however, judging from the results obtained by determining the solubility of the variously produced hard waxes in hot acetone followed by hot ethanol. It is believed that a measure of solubility of the waxes in these solvents is quite indicative of the variations in composition of the hard waxes. The data obtained in this connection are given in Table 10, and it may be noted that there is little difference in the total of the acetone soluble and ethanol-soluble portions of hard waxes found by the use of acetone in comparison with 2-butanone for removing the fatty fraction. However, there is a distinct difference in the total of soluble portions found by the use of toluene compared with petroleum naphtha. The hard wax derived from naphtha-extracted crude wax contains a distinctly

smaller proportion of acetone-soluble plus ethanol-soluble matter. This fact would be of particular significance if one attempted to produce a light-colored wax to meet special requirements, because successful isolation of light-colored sugarcane wax appears to be possible only through fractionation of the dark hard wax by means of solvents. In order to obtain the highest yield of light-colored wax, toluene is preferred over petroleum naphtha for extracting the crude wax.

Apparently most of the color of the hard wax remaining after removing the fatty matter which carries most of the chlorophyll and other fat-soluble pigments is associated with the fraction of the hard wax that is insoluble in boiling ethanol alone or in acetone followed by ethanol. This insoluble fraction has such distinctly different properties from the soluble fraction that it must differ materially from the latter in composition. Aside from being extremely dark, it becomes very brittle upon cooling; also it contains most of the ash-forming substance extracted from the crude wax. The presence of this fraction contributes to the hardness and brittleness of the original hard wax. The production of a light-colored wax is dependent upon the removal of this very dark fraction for it appears to be unbleachable by any known method; this is most easily accomplished by means of some solvent to extract the light-colored fraction and leave the dark fraction as an insoluble residue.

Table. 9
Acetone vs. 2-Butanone for Extraction of Fatty Matter from Crude Wax-Percentage of Hard wax on basis of total crude wax.

Source of Wax	Toluene Extracted Crude Wax		Naptha Extracted Crude Wax	
	Acetone	Butanone	Acetone	Butanone
	%	%	%	%
1-1941	79.5	64.7	----	----
1-1942	73.7	62.5	86.3	80.3
2-1942	70.1	69.5	76.1	----
4-1942	71.2	65.2	70.3	65.9
5-1942	75.0	72.9	----	----
6-1941	72.1	72.9	79.1	72.3
Ave.	73.6	68.0	78.0	72.8

In selecting a solvent for effecting this separation, it was desired to find one which would give a maximum yield of the light-colored fraction without dissolving any of the very dark fraction and one from which it would be possible to obtain the wax in a suitably crystalline form for easy separation of the mother liquor for highest purification of the wax. In respect to yield of light-colored wax, best results were obtained from 2-propanol (iso-propyl alcohol) but ethanol almost equalled its performance. Both of these solvents should be anhydrous for best results. The extraction may be easily made by digesting the hard wax, after the removal of the fatty matter, with about 10 times its weight of alcohol under a reflux condenser until equilibrium is established. Stirring aids the operation. Acetone and methanol yielded a lesser quantity of light-colored wax than ethanol, as would be expected. Butanol, ethyl acetate and other solvents that completely dissolve the hard wax, including the undesirable fraction, may be used if their solvent action is lowered with water or some other dilutant. However, none has yet been found to be quite as satisfactory as 2-propanol.

Any chlorophyll that is retained in the hard wax will be extracted with the light-colored fraction by the alcohol. This may be removed from the extracted light-colored wax fraction by treating the alcohol solution with a decolorizing carbon in a customary manner. As already suggested, further refinement of the wax may be effected by allowing the way to crystallize from the decolorized wax solution and removing the mother liquor by filtration or other physical means. Sugarcane wax having a color equal to No. 1 carnauba wax has been obtained by such a method.

It will be observed from Table 10 that the combined acetone and ethanol fraction is about what may be expected with ethanol or with 2-propanol alone. It may be noted that the yield of light-colored wax, if derived from toluene-extracted crude wax, will average about 78% of the dark hard wax. This applies only to waxes of Louisiana origin, however. Peculiarly, waxes of other origin will not yield such a high proportion of the light-colored fraction nor yield a light-colored wax equaling the Louisiana wax in hardness. Waxes from Cuba, South Africa and South America prepared similarly to the Louisiana waxes have been examined. It may be concluded that the composition of Louisiana wax differs quite appreciably from waxes of other sources even though receiving similar treatment.

In Table 10 are also reported other data regarding properties of hard Louisiana waxes produced under pilot plant conditions. The colorimetric readings of solutions of the various lots of waxes definitely shows that more color is extracted from toluene than with naphtha and that 2-butanone more efficiently removes color than acetone in the process of extracting the fatty fraction from crude wax. The variation in the ash content of the hard wax is due mostly to the variation in the composition of the mud press cake for, on a given sample of muds, crude waxes having practically identical ash contents were obtained with toluene, benzene, petroleum naphtha and carbon tetrachloride. Analysis of the fatty fractions for ash contents shows that 2-butanone extracts a higher proportion of ash forming constituents from crude wax than does acetone. As already stated, the ash is associated with the extremely dark-colored fraction of the hard wax which is insoluble in boiling ethanol; the light colored fraction soluble in ethanol generally contains only about 1/10th the ash of the original hard wax. It has not yet been determined what role the ash plays or how it may be combined. Its proportion can be reduced in the wax by means of an acid treatment but such treatment does not increase the proportion of ethanol-soluble matter. It might be concluded from this that the insolubility of the dark-colored fraction is not dependent upon some sort of combination of the metals and organic acids. About 50% of the ash was found to consist of calcium, iron and aluminum oxides. Some of the iron is certain to be the result of contamination from pilot plant equipment but most of it, as well as the calcium and aluminum, must have found its way into the muds as a result of elmination from the juices in the clarification process.

The specific gravity of hard sugarcane wax as produced by the two solvent method is very close to 1.0. Apparently the ash content influences the specific gravity to some extent as there is a tendency for waxes of high ash content to have the higher specific gravity.

Some laboratory work has been done toward finding a suitable method of fractionating the fatty matter extracted with acetone or 2-butanone from crude wax for the production of valuable products. The conclusion was reached that the most satisfactory